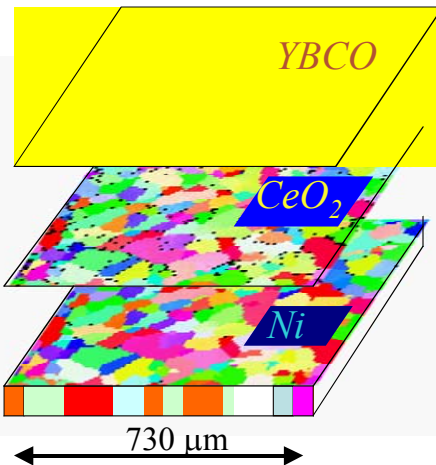


Science With Microbeams

APS Science Advisory Committee
Cross-Cut Review

January 21, 2004

Introduction, J. Murray Gibson



Purpose of the Review

- Science Advisory Committee does sector-by-sector reviews
- Cross-cut gives an important perspective
 - focus of review is retrospective (“where are we now”)
 - additional experts invited
- Report will advise APS on strengths and opportunities
- Session is open for healthy information exchange

How did we select the program?

- Current research programs involving x-rays focused to $< 10\mu\text{m}$
- Asked sectors for their best science
- Only one presentation per sector, due to time
 - Sectors included based on scale of activity
- Further information provided as backup
 - to be posted on the web

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(Murray Gibson)

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(Guoyin Shen - Sector 13)

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microscale level at GSECARS**
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Future of Nanobeam Studies**
(Eric Isaacs)

Cross-Cut Review: Science with Microbeams

Gerhard Materlik, Chair

Program

Wednesday, January 21, 2004

Bldg. 402 Conference Center

Advanced Photon Source

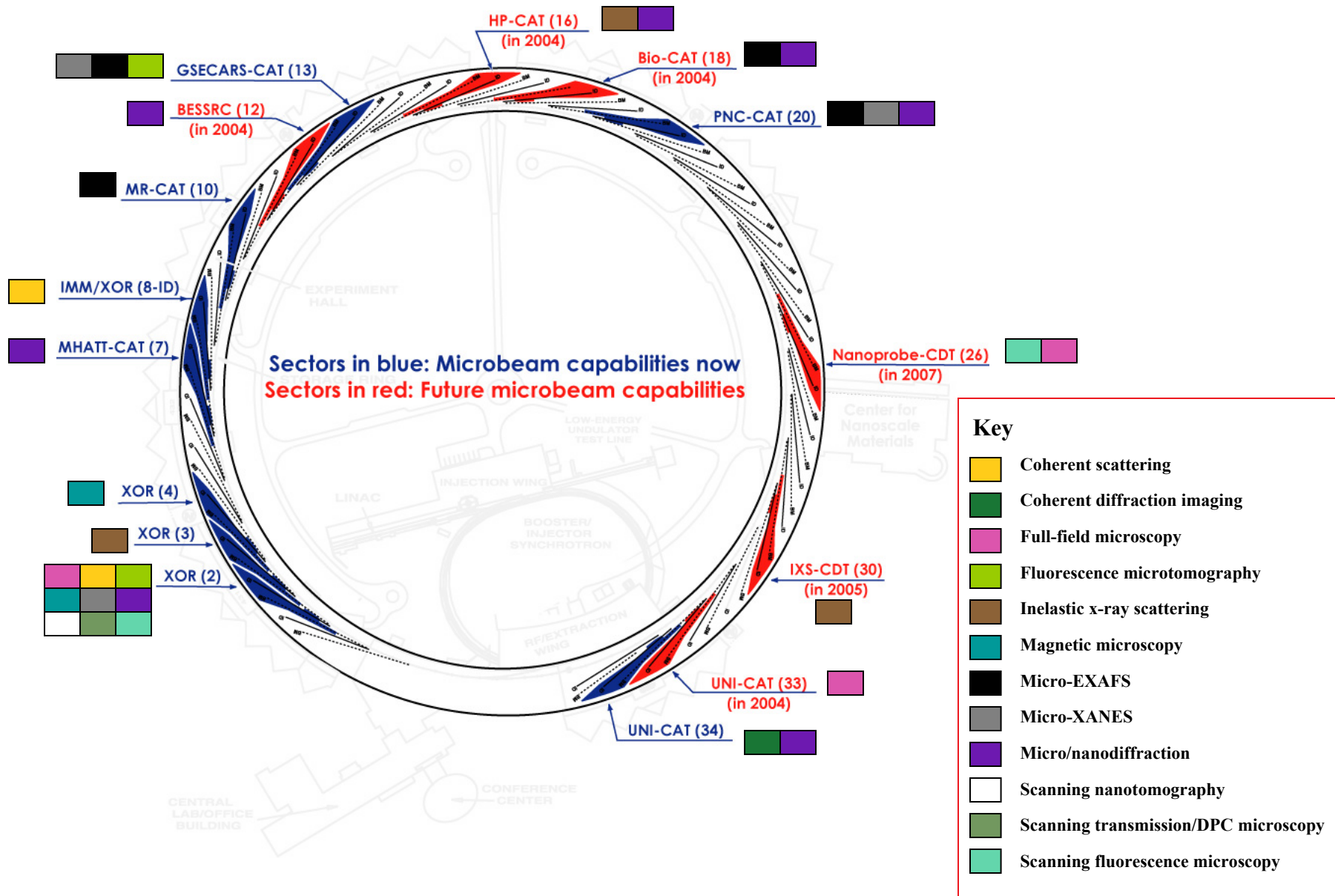
Argonne National Laboratory

Center for
Nanoscale
Materials

**9:00 a.m.: Three-dimensional polychromatic microdiffraction
studies of mesoscale structure and dynamics**
(Gene Ice - Sector 34)

**9:35 a.m.: Microbeam imaging of crystals by coherent
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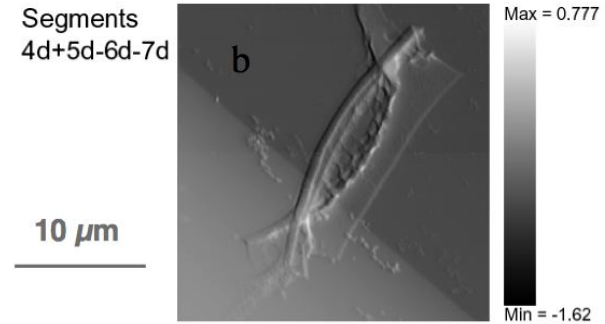
APS X-ray Microbeam Techniques



X-ray microbeam techniques in use at APS

Scanning transmission/DPC microscopy

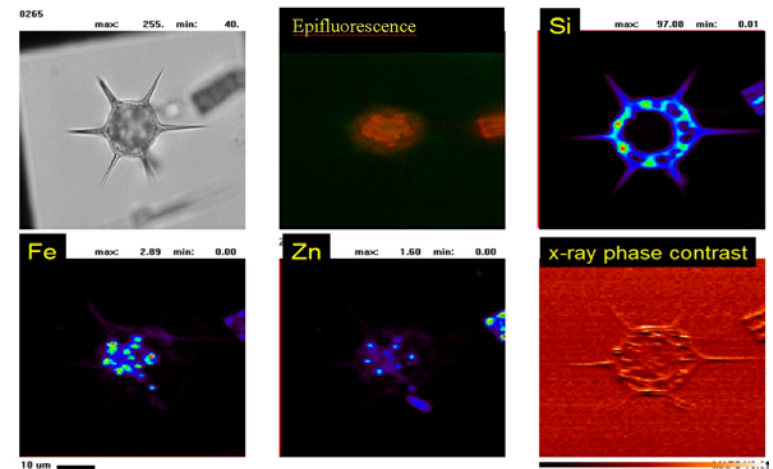
X-ray optics are used to form a microfocused spot on the sample, which is raster-scanned to form images in absorption and differential phase contrast modes. STXM is fast and can be combined with XANES as for SFXM.



Scanning differential phase contrast image of a diatom, taken with 1.8 keV x-rays at 2-ID-B.

Scanning fluorescence microscopy

X-ray optics are used to form a microfocused spot on the sample, which is raster-scanned to form images in x-ray fluorescence contrast. SFXM is highly sensitive to elemental makeup and chemical state information.

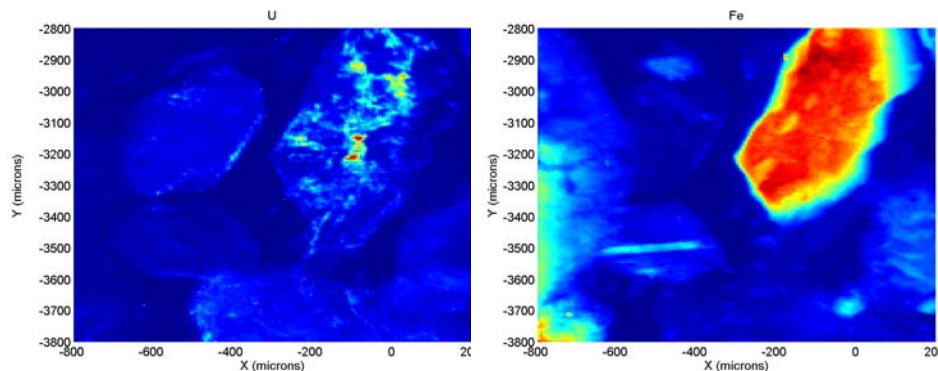


Visible light and epifluorescence micrographs, false-color SFXM element maps, and x-ray phase contrast image of a silicoflagellate from the Southern Ocean. Data from 2-ID-E.

X-ray microbeam techniques in use at APS

Micro-EXAFS

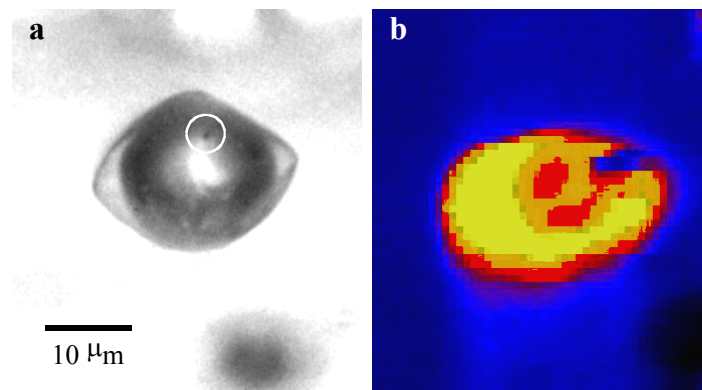
Similar to SFXM, but incident energy is scanned over a wide range to obtain extended x-ray absorption fine-structure spectra (usually fluorescence mode) at each sample point. Spectra are Fourier-inverted to discern atomic structure.



U and Fe fluorescence maps taken at 20-ID from a sediment thin section from beneath a leaking rad-waste storage tank at Hanford.

Micro-XANES

Similar to SFXM, but incident energy is scanned in fine steps to obtain x-ray absorption near-edge spectra (e.g. by fluorescence) at each sample point.



(a) Optical and (b) Cu K_{α} images of a vapor phase fluid inclusion in quartz taken at 13-ID, showing that Cu is uniformly distributed in the fluid phase. Cu K micro-XANES spectra show reversible changes in Cu oxidation with temperature. This may control speciation behavior during formation of important Cu deposits.

X-ray microbeam techniques in use at APS

Full-field microscopy

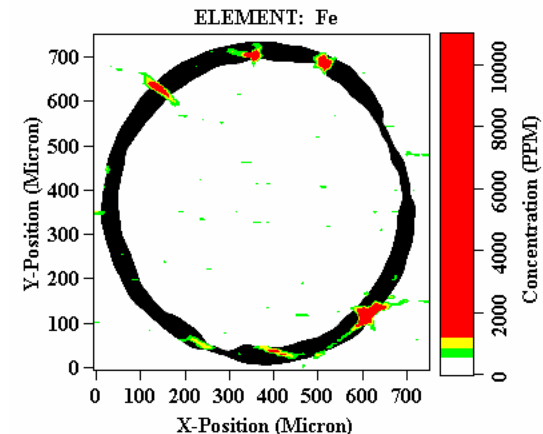
High resolution x-ray optics are used to image the sample directly onto an array detector such as a CCD camera. Main advantages are speed and amenability to phase contrast methods.



Quantitative 3D reconstructions of the real part of the refractive index of a silicon atomic force microscope tip. Coherent full-field projections taken at 2-ID-B.

Fluorescence microtomography

Similar to SFXM, but sample is rotated through a large angular range to record many views of it. Projection data are filtered and reconstructed to form 3D images of the sample.

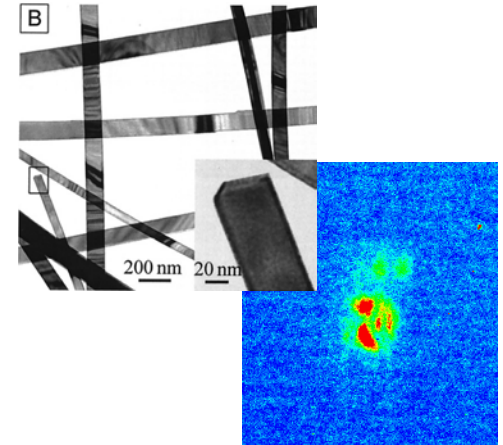


Reconstructed slice showing trace Fe content in a SiC nuclear fuel shell, using Fe K fluorescence microtomography data recorded at 2-ID-D.

X-ray microbeam techniques in use at APS

Micro/nanodiffraction

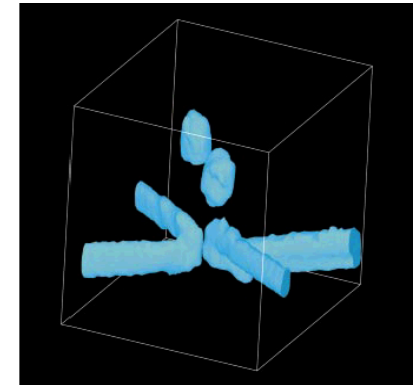
Microfocusing optics such as KB mirrors and zone plates are used to isolate specific sample regions and increase the x-ray flux density in diffraction experiments.



SEM images of Sn₂O₃ nanobelts. X-ray diffraction pattern from a 30 nm x 10 nm nanobelt, recorded at 2-ID-D.

Scanning nanotomography

Similar to STXM, but sample is rotated through a large angular range to record many views through it. Projection data are filtered and reconstructed to form 3D images of the sample.

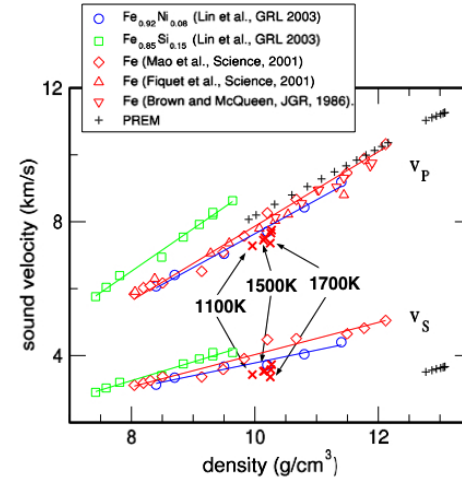


Reconstruction of 13 projections (1573 eV) through two-level chip sample, showing W vias and Al interconnects in (8 μm)³ volume. Data from 2-ID-B.

X-ray microbeam techniques in use at APS

Inelastic x-ray scattering

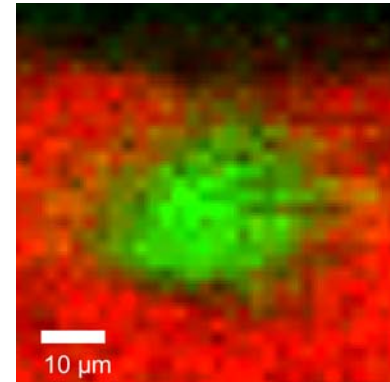
Microfocusing optics such as KB mirrors are used to increase the x-ray flux density on the sample in inelastic scattering experiments.



Temperature effect on sound velocities in iron at high pressure measured at room temperature (except red crosses) at 3-ID-B.

Magnetic microscopy

Similar to SFXM, but incident x-ray beam is linearly or circularly polarized to form images of the sample with magnetic charge contrast by absorption or diffraction methods.

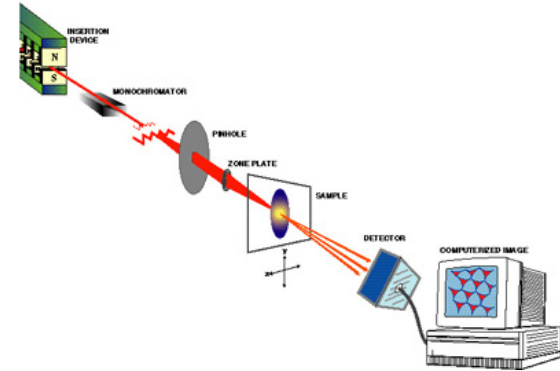


Magnetic diffraction contrast image of superposed ($\delta, 0, 1$) (red) and $(0, \delta, 1)$ (green) incommensurate reflections from spin density wave domains in a Cr single crystal at 110 K. Data recorded at 2-ID-D.

X-ray microbeam techniques in use at APS

Coherent scattering/speckle

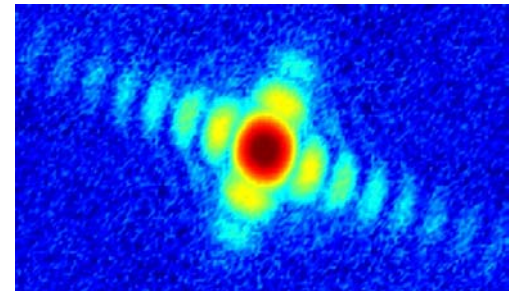
Microfocusing optics such as zone plates are used to tailor the x-ray beam phase space to the sample in coherent scattering and speckle experiments.



Coherent x-ray scattering experiment using zone plate microfocusing optics, as at 2-ID and 8-ID.

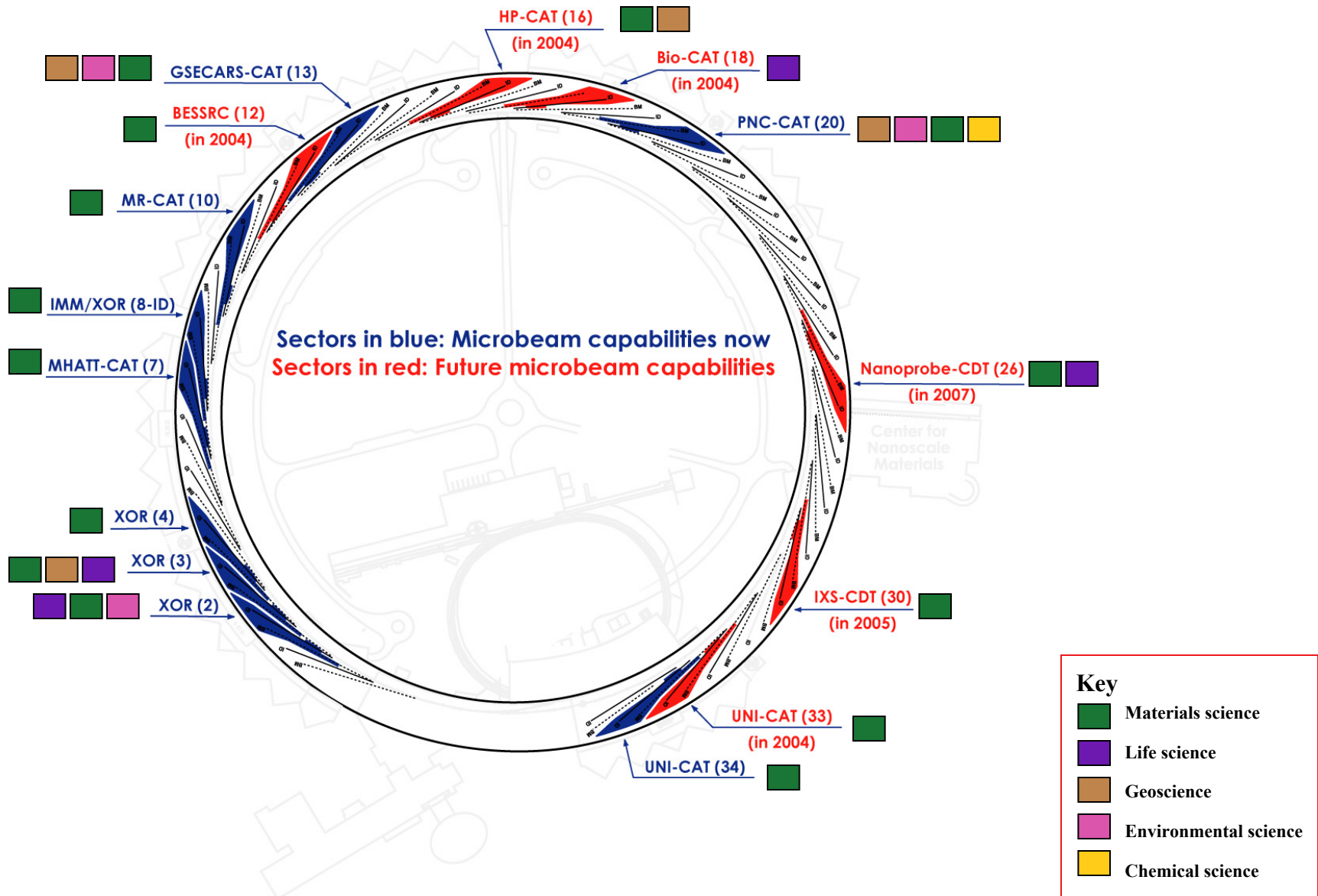
Coherent diffraction imaging

Microfocusing optics such as KB mirrors are used to increase the x-ray flux density on the sample in coherent diffraction imaging experiments.

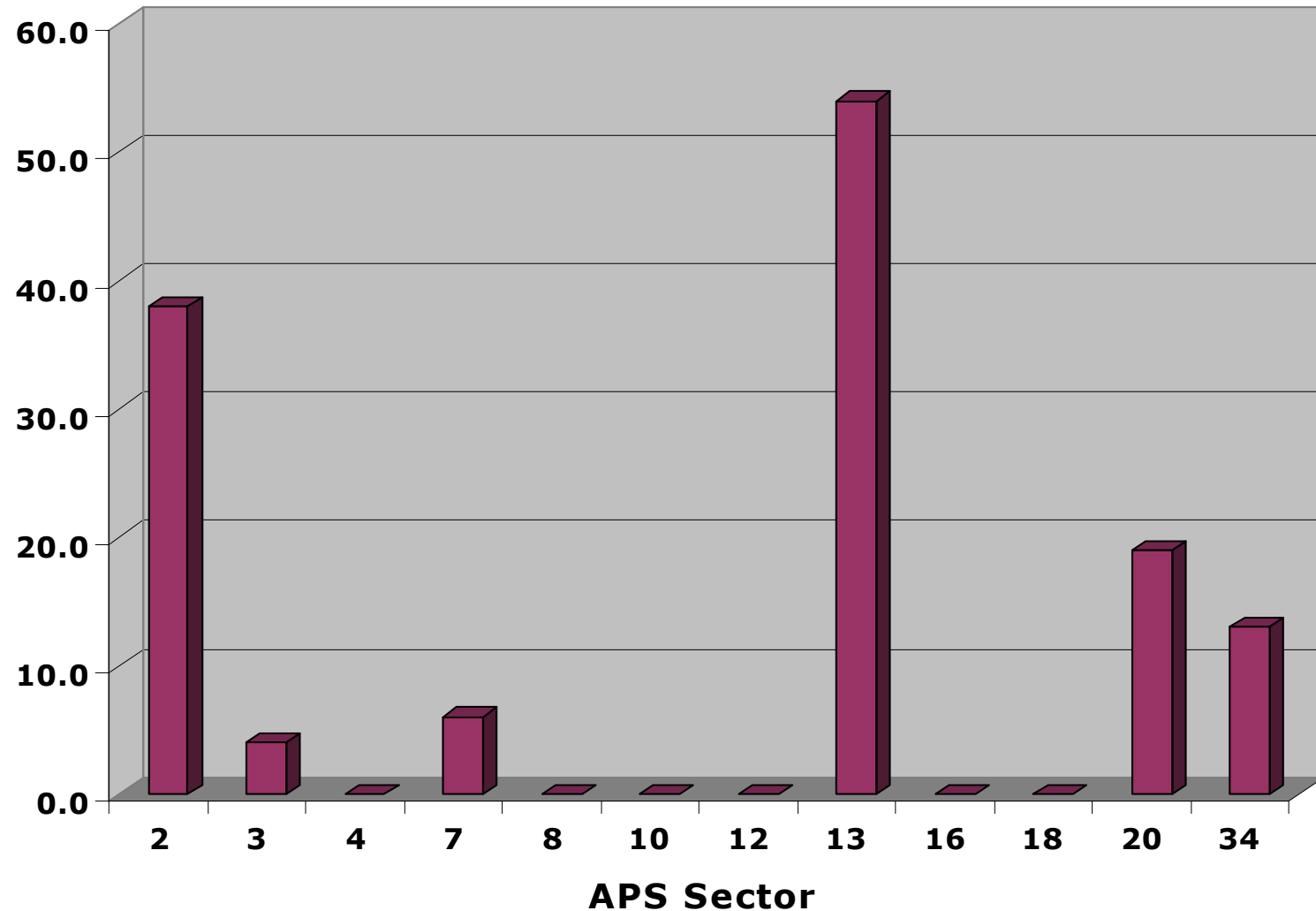


Coherent x-ray diffraction pattern of a single silver nanocube obtained by chemical synthesis, recorded at 34-ID-C.

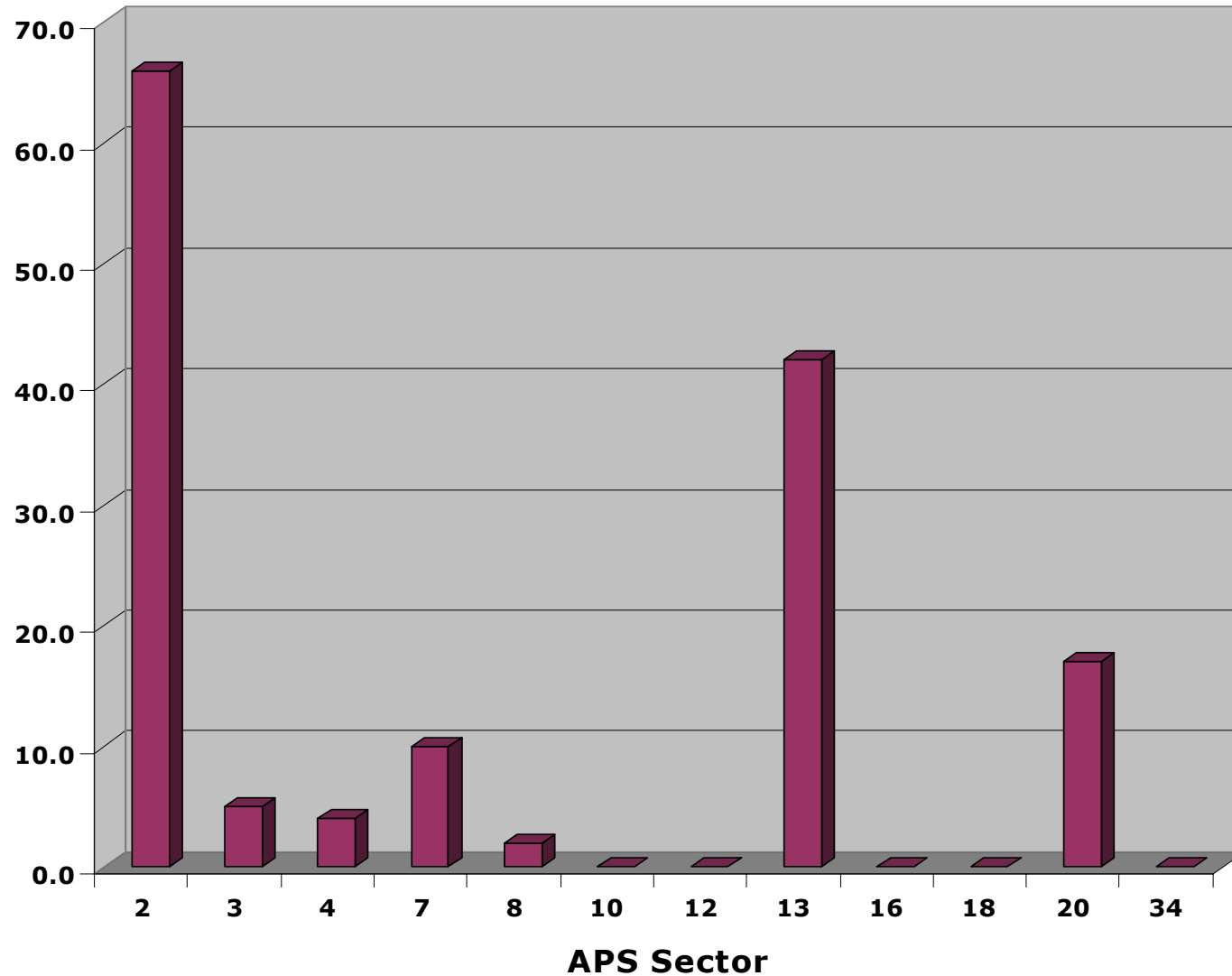
APS X-ray Microbeam Applications by Scientific Discipline



APS microbeam publications by sector (2002-2003)



Allocated GUPs by sector (2003)



New microbeam initiatives

- **Nanoprobe beamline (Nano-CDT, 26-ID)**
 - Funded, MOU signed, construction underway
- **Inelastic x-ray scattering beamline (IXS-CDT, 30-ID)**
 - Funded, MOU signed, construction underway
- **Micro XAS and Diffraction capability at BIOCAT**
 - Under development (operational 2005)
- **Environmental microspectroscopy beamline (EnviroCAT)**
 - Proposed and partially-funded
- **Multiplexed microfocusing beamline (UNI-CAT, 34-BM)**
 - Partner User Proposal Letter of Intent submitted
- **1-4 keV coherent imaging/scattering beamline (XOR)**
 - Partner User Proposal in preparation
- **Microfocus x-ray crystallography**
 - Needed at APS (UC Review Committee/Janet Smith)

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